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Indian Standard

SYNTAN FOR PREVENTION OF SLUDGE FORMATION IN VEGETABLE TAN LIQUORS— SPECIFICATION

UDC 675·042

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

April 1991

Price Group 3

Tanning Materials and Allied Products Sectional Committee, CHD 18

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 27 July 1990, after the draft finalized by the Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

This type of syntan is largely used in vegetable tanning industry mainly for clarifications and prevention of sludge formation in natural tannin liquors during tanning and vegetable tannin extract manufacture. In this way, it minimises the loss of tannin and sludge formation. Normally, this syntan is blended with natural tannin at appropriate stages during extract manufacture or tanning. Since this syntan is not meant for self tanning, tannin content is, therefore, not essential.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SYNTAN FOR PREVENTION OF SLUDGE FORMATION IN VEGETABLE TAN LIQUORS — SPECIFICATION

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for syntan for prevention of sludge formation in vegetable tan liquors.

2 REFERENCES

2.1 The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
265 : 1987	Specification for hydrochloric acid (*third revision*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
1640 : 1960	Glossary of terms relating to hides, skins and leather
4905 : 1968	Methods for random sampling

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 1640 : 1960 shall be followed.

4 REQUIREMENTS

4.1 The material shall be in the form of powder or liquid.

4.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

5 PERFORMANCE TEST

The sludge formation shall not be more than one percent by mass, when tested according to the method prescribed in Annex B.

6 PACKING AND MARKING

6.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be packed in moisture proof containers like gunny bags suitably lined with moisture proof paper or polyethylene bags.

6.2 Marking

The containers shall be securely closed and bear legibly and indelibly the following particulars:

a) Name of the material,
b) Indication of the source of manufacture,
c) Gross and net mass,
d) Date of manufacture, and
e) Batch No.

7 SAMPLING

7.1 Preparation of Test Samples

Representative test samples of the material shall be prepared as prescribed in Annex C.

7.2 Number of Tests

Tests for the determination of chemical requirements shall be conducted on the composite sample.

7.3 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the standard, if all the test results for the tests conducted on the composite samples meet the relevant stipulations for the different characteristics.

Table 1 Requirements of Syntan for Prevention of Sludge Formation in Vegetable Tan Liquors
(*Clause* 4.2)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Cl No. in Annex A
(1)	(2)	(3)		(4)
i)	Moisture, percent by mass, *Max*	Powder Liquid	5·00 50·00	A-4
ii)	Total solid residue, percent by mass, *Min*	Powder Liquid	95·00 50·00	A-5
iii)	*p*H of 10 percent solution		6·5 to 7·5	A-6
iv)	Iron (as Fe_2O_3), percent by mass, *Max*		0·03	A-7
v)	Ash, percent by mass, *Max*		30·00	A-8

ANNEX A

(*Clause* 4.2, and *Table* 1)

METHODS OF TEST FOR SYNTAN FOR PREVENTION OF SLUDGE FORMATION IN VEGETABLE TAN LIQUORS

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used for the tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

A-2 PREPARATION OF SAMPLE FOR TEST

A-2.1 Spray Dried Powder

Mix thoroughly and sample.

A-2.2 Liquid Syntan

Mix thoroughly so as to include any sediment which may be present in the liquid sample.

A-3 PREPARATION OF SOLUTION

A-3.1 Quantity of the Material

Weigh out such a quantity of the material which shall give as nearly as possible 4·0 g per litre of tannin matter absorbable by hide powder, but in any case not less than 3·75 g and not more than 4·25 g.

NOTE — In the event of the results of an analysis showing a tanning strength outside these limits, the analysis shall be repeated employing the required amount of material.

A-3.2 Solid Syntan

Weigh out such a quantity of the material which will give as nearly as possible 1 litre of solution of required analytical strength (*see* **A-3.1**). Add the material into a beaker containing boiling water, stir, and transfer the dissolved portion to a 1 000-ml flask. Repeat the process until all the material is dissolved and the flask contains about 500 ml of solution. Cool and make up the volume.

NOTE — Syntans which are sensitive to boiling water should be dissolved at a lower temperature, and the temperature shall be stated in the report.

A-3.3 Liquid Syntans

Weigh out such a quantity of the material which shall give as nearly as possible 1 litre of solution of required analytical strength (*see* **A-3.1**). Add 400 ml boiling water and transfer to a 1 000-ml graduated flask. Add further boiling water to bring the volume to about 900 ml. Cool and make up the volume.

A-3.4 In the estimation of total solids, total solubles and non-tannins, the aliquots shall be maintained at *p*H 7.0 before evaporation over steam bath by the addition of required quantities of dilute standard sodium hydroxide solution. The necessary correction for the sodium hydroxide added should be applied to the mass of the dry residues while making calculations to determine the analytical characteristics.

A-4 DETERMINATION OF MOISTURE

A-4.1 Apparatus

A-4.1.1 *Air Oven*

With thermostatic control, capable of maintaining 100 ± 2°C.

A-4.1.2 *Wide-Mouthed Weighing Bottle*

A-4.2 Procedure

Transfer about 2 to 5 g of the finely ground material (*see* **A-2**) into a tared wide-mouthed weighing bottle and weigh accurately. Dry it at 98·5 to 100°C in an oven for 3 to 4 hours, cool in a desiccator for about 20 minutes and weigh again accurately. Repeat the process of drying and weighing until two weighings at an interval of one hour do not differ by more than 0·2 mg.

NOTES

1 Since for any syntan the sum of the moisture and the total solids is 100 percent, the determination of either is sufficient.

2 If the weight of the dried material is found to increase with prolonged drying, then the lowest weight found shall be taken.

A-4.3 Calculation

A-4.3.1 *Solid Syntan*

$$\text{Moisture, percent by mass} = \frac{(M_1 - M_2) \times 100}{M_1}$$

where

M_1 = mass in g of the material taken for the test, and

M_2 = mass in g of the residue left after drying.

A-4.3.2 *Liquid Syntan*

$$\text{Moisture, percent by mass} = 100 - X$$

where

X = total solid (*see* **A-5**).

A-5 DETERMINATION OF TOTAL SOLID

A-5.1 Apparatus

A-5.1.1 *Steam-Bath*

A-5.1.2 *Air Oven*

With thermostatic control, capable of maintaining 100 ± 2°C.

A-5.2 Procedure

Evaporate to dryness, on a steam bath, 50 ml of the syntan solution prepared from a known amount of the material, according to the method prescribed in **A-3**. Dry the residue at 98·5 to 100°C in an oven to a constant mass. Consider the mass as constant when the two weighings, at an interval of one hour, do not differ by more than 0·2 mg.

A-5.3 Calculation

$$\text{Total solid, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_2 = mass in g of the material left after drying, and

M_1 = mass in g of the material taken for test.

A-6 DETERMINATION OF *p*H

A-6.1 Apparatus

A-6.1.1 p*H Meter* — with glass electrode.

A-6.2 Procedure

Determine the *p*H of the solution prepared in **A-3** adjusting the relative density to 1·05 at 20°C with cold water, by using a suitable *p*H meter.

A-7 DETERMINATION OF IRON

A-7.1 Reagents

A-7.1.1 *Fuming Nitric Acid*

A-7.1.2 *Bromine Water* — saturated.

A-7.1.3 *Concentrated Hydrochloric Acid* — (*see* IS 265 : 1987).

A-7.1.4 *Ammonium Hydroxide* — 1 : 1 (*v/v*).

A-7.1.5 *Dilute Hydrochloric Acid* — 1 : 1 (*v/v*).

A-7.1.6 *Dilute Sulphuric Acid* — 1 : 20 (*v/v*).

A-7.1.7 *Potassium Permanganate Solution* — approximately 0·1 *N*.

A-7.1.8 *Postassium Thiocyanate Solution*

Dissolve 10 g of potassium thiocyanate (or ammonium thiocynate) in 100 ml of mater

A-7.1.9 *Stock Iron Solution*

Dissolve exactly 0·702 g of pure ferrous ammonium sulphate [$FeSO_4$, $(NH_4)_2SO_4$, $6H_2O$] in 50 ml of water and 20 ml of dilute sulphuric acid (1 : 4) and add 0·1 *N* potassium permanganate solution dropwise until a faint pink colour is obtained. Dilute the solution to 1 000 ml with water. The strength of this solution is ten times that of the standard solution. The keeping quality of this solution is better at this strength. Store this solution and use when required.

A-7.1.10 *Standard Iron Solution*

Prepare a fresh solution by diluting 10 ml of the stock solution (**A-7.1.9**) to 100 ml with water. Each millilitre of this solution contains 0·000 01g of iron (as Fe).

A-7.2 Procedure

A-7.2.1 Weigh accurately a quantity of the material, which is equivalent to 5 g on moisture-free basis, to the nearest 0·1 mg in a glazed flat-bottomed porcelain basin. Heat it over a low flame until the residue is completely charred and ceases to give out smoke. Further char the sample in a muffle furnace at 600°C to make it free of carbon.

A-7.2.2 In case all the carbon is not removed, take out the basin from the furnace and allow it to cool. Add a little hot water and break up the carbonaceous matter with a glass rod. Extract it with water by warming it over a water-bath for a few minutes. Decant and collect the filtrate. Repeat the process of extraction and filtration three times. Combine the filtrates. Transfer the residue on the filter paper and wash it several times with hot water and add the washings to the filtrate. Place the filter paper and the contents in the original basin, dry, ignite and ash as before, until all the carbon is burnt off. Cool the basin again and transfer the combined filtrates and washings quantitatively to it. Evaporate the extract to dryness on a water-bath. Replace the basin in a muffle furnace (600°C).

A-7.2.3 Cool the basin and moisten the carbon-free ash with hot water. To this add five drops each of fuming nitric acid and bromine water. Heat slowly until the fumes cease to come out. Evaporate to dryness. Moisten the residue with water and digest it with 5 ml of concentrated hydrochloric acid over a water-bath for a few minutes, dilute with water and filter into a 250 ml beaker. Adjust the volume to approximately 75 ml (by boiling, if necessary) and make it faintly alkaline with ammonium hydroxide. Boil off the excess ammonia gently and allow the precipitated iron and aluminium hydroxides to coagulate.

A-7.2.4 Allow the solution to stand for few minutes until the precipitate settles. Decant the supernatant liquid quickly through an asbestos Gooch filter, previously washed with hot dilute hydrochloric acid, water, ammonium hydroxide and water successively. Wash the precipitate three times with hot water made faintly alkaline with five drops of ammonium hydroxide per litre. Transfer the precipitate quantitatively to the filter paper and wash it four times with hot, faintly ammoniacal water.

A-7.2.5 Transfer 10 ml of dilute sulphuric acid and 5 ml of potassium thiocyanate solution to each of the two identical 50 ml Nessler cylinders. To one of the Nessler cylinders add 10 ml of the made up solution (*see* A-7.2.3) and make up the volume to the mark. From a burette, run in standard iron solution to the second Nessler cylinder until the tints of the two cylinders are identical when viewed vertically through the depths of the cylinders, the cylinders being held over a white surface.

A-7.3 Calculation

Calculate the iron content of the sample from the known volume of the standard iron solution required for matching.

A-8 DETERMINATION OF ASH

A-8.1 Procedure

Heat gently about 2 g of syntan in a silica crucible on a Bunsen burner till ashed. Transfer the crucible to a muffle furnace maintained at 800°C and heat for three hours. Repeat the process till constant mass is obtained.

A-8.2 Calculation

$$\text{Ash, percent by mass} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass in g of the residue obtained, and

M = mass in g of the sample taken for test.

ANNEX B

(*Clause* 5)

PERFORMANCE TEST FOR SYNTAN FOR PREVENTION OF SLUDGE FORMATION IN VEGETABLE TAN LIQUORS

B-1 PROCEDURE

B-1.1 Prepare a mixture of vegetable tannin extract and syntan in the ratio 95 : 5 (on solid basis) and dissolve in distilled water to make a 10 percent (*m/v*) solution. Separately prepare solutions of vegetable tannin and syntan of the same concentration (10 percent, *m/v*) in distilled water.

B-1.2 Take 100 ml of each of the solutions prepared in **B-1.1** in separate measuring cylinders, shake and leave for 6 h at room temperature to settle. Record the amount of sludge/sediments settling after 6 h. The sludge formed shall not be more than 1 percent.

ANNEX C

(*Clause* 7.1)

PREPARATION OF TEST SAMPLES OF SYNTAN FOR PREVENTION OF SLUDGE FORMATION IN VEGETABLE TAN LIQUORS

C-1 SCALE OF SAMPLING

C-1.1 Lot

In any consignment, all the containers of the material of the same size and drawn from a single batch of manufacture shall constitute a lot. If a consignment is known to consist of different batches of manufacture or of different sizes of containers, the containers belonging to the same batch and sizes shall be grouped together and each such group shall constitute a separate lot.

C-1.2 For ascertaining the conformity of the lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose (*n*) shall depend on the size of the lot (*N*) and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

(*Clause* C-1.2)

Lot Size (*N*)	No. of Containers To be Selected (*n*)
Up to 100	3
101 to 300	4
301 and above	5

C-1.3 The containers shall be selected at random from the lot and in order to ensure randomness of selection, use of random number tables (*see* IS 4905 : 1968) shall be made. In case random number tables are not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, ..., up to r and so on, r being the integral part of N/n, where N is the lot size and n the number of containers to be selected. Every rth container thus counted shall be withdrawn to give the sample for tests.

C-2 PROCEDURE

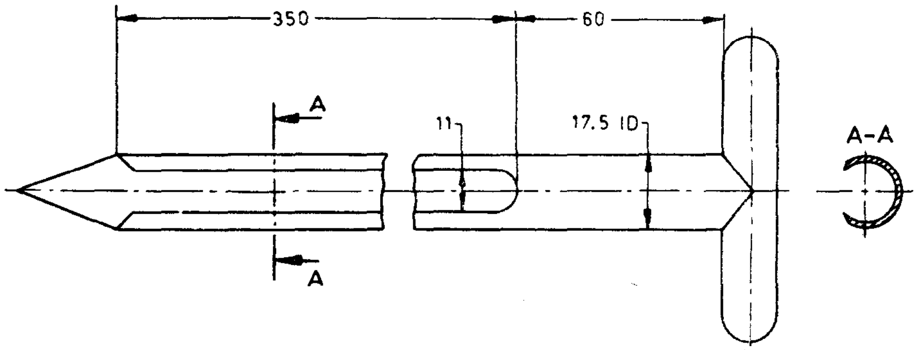
C-2.1 For taking cut material, the containers shall be opened and the material taken out with the help of a sampling tool shown in Fig. 1 or any other suitable appliance. From each of the containers selected according to **C-1.3**, small portions of the material shall be drawn from different parts so to get a representative sample. The total quantity of the material so collected shall be sufficient to carry out the tests as required in **4** and **5**.

C-2.2 Out of these portions, a small but equal quantity of the material shall be taken out and thoroughly mixed to form a composite sample having a mass of about 200 g. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

C-2.3 All the composite samples shall be immediately transferred to separate containers, and sealed airtight and labelled with full identification particulars, such as manufacturer's name or trade-mark, month and year of manufacture of the material, batch numbers (if available), date of sampling, sampler's name, etc.

NOTE — The time taken from opening of the original containers to the sealing of samples shall be as short as possible. Care shall be taken to protect the sample from moisture and other adventitious contamination.

C-2.4 The referee composite test sample shall bear the seal of both the purchaser and the supplier. They shall be kept at a place till such time as agreed to between the purchaser and the supplier to be used in case of any dispute between the two.



All dimensions in millimetres.

FIG. 1 SAMPLING TOOL

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Doc : No. CHD 18 (9383)

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Swatantra Bharat Press, Delhi, India